

Miniature Quartz Crystal Microbalance for Spacecraft and Missile Applications

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Quartz crystal microbalances (QCMs) have been used for over 20 years as contamination monitors in spacecraft to measure film deposition on sensitive surfaces such as optical mirrors, thermal radiators, and solar arrays. Only recently, however, have miniature QCMs been used in a cryogenically cooled infrared space telescope and in the seeker of a missile to measure optical contamination from outgassing materials. This article describes a missile application in which we coated a QCM with low outgassing hydrocarbon grease to trap and measure the mass of particles impinging on its surface. We also report the results of several experiments using different QCM sensors for both spacecraft and missile use. (Keywords: Contamination, Missile, MSX, Quartz crystal microbalance, Space.)

INTRODUCTION

The use of quartz crystal microbalances (QCMs) to measure and detect condensable materials in space is well documented.¹⁻³ During the early 1990s, the Mid-course Space Experiment (MSX) was tasked to quantify the effect of contamination on the optical performance of the Space Infrared Imager and Telescope (SPIRIT III) for the entire mission.⁴ To accomplish the task, knowledge of the amount of contamination on the SPIRIT III primary mirror in space was required. The best way to measure the contamination was to place a contamination sensor next to the primary mirror, but not within its field of view. Unfortunately, the primary mirror was deep inside the telescope baffles and space was limited. In addition, the sensor would have to operate at the extremely cold temperature of the mirror, which was expected to be 20 K.⁴

APL supports the Navy in the development of the Standard Missile-Block 3 (SM-3). Last year, we performed aerothermal tests of its nose cone in the Avery Advanced Technology Demonstration Laboratory (AATDL) wind tunnel facilities. Initial tests indicated potential contamination that could affect the performance of the seeker optics.⁵ To reduce the risk of contamination, several materials and design modifications were made to the nose cone. Instruments including optical witness samplers as well as two QCMs were added to monitor the outgassing of the nose cone during the next series of tests. In addition, a contamination cover on the seeker optics was added to assess its protective value.

Besides being flight qualified, the QCMs for the missile tests had to be small enough to fit within the

confines of the existing SM-3 nose cone and sensitive enough to measure the contamination level required by the mission. Primarily on the basis of our experience with MSX, we designed, fabricated, and qualified the Contamination Sensor System (CSS) to meet SM-3 program requirements as developed in an interface control document (ICD)⁶ negotiated with the prime contractor.

The first part of this article describes the rationale, design, and results to date of this innovative QCM sensor as used in the MSX spacecraft and in the missile nose cone.

QCM OPERATIONS AND DATA ANALYSIS

Knowledge of the operation of QCMs (Fig. 1) is essential to understanding the results of the space and missile experiments describe here. Their operation is related to the optical quantities to be measured, e.g., the bidirectional reflectance diffusion function (BRDF) of the SPIRIT III telescope or the reflectance or transmission of the missile seeker optics due to either particulate contamination or molecular film deposition.

The QCM uses two carefully matched quartz crystals, one under the other; only one, however, is exposed to the outside environment. The difference in

frequency between the two crystals, i.e., the beat frequency, is a very sensitive indication of the mass being deposited on the exposed crystal surface. These QCMs can measure contamination with angstrom thickness resolutions, clearly less than a monolayer resolution.² The beat frequency is proportional to the mass of contamination that has accumulated on the sensing area and is electronically recorded in a digital electronic counter. The counter is updated at a rate sufficient to resolve the dynamics of the contamination history. For the SM-3 system,⁶ we provided the QCM data output in analog form (0 to 5 V DC) to simplify the interface to the existing SM-3 missile telemetry.

In the CSS signal conditioner module, the digital count from the QCMs is converted using a digital-to-analog converter for each QCM channel. The converters are updated at a rate sufficient to minimize switching errors. In addition, the controller injects a calibration-count word sequence to the input of the converters once every second to provide a recognizable end-to-end calibration for the QCM analog signal. During data analysis of the received telemetry signals, the calibration count permits reconstruction of the actual QCM frequency measured. This measured frequency is then corrected for shifts due to temperature using the specific QCM temperature-to-frequency calibration, which is unique to each sensor.

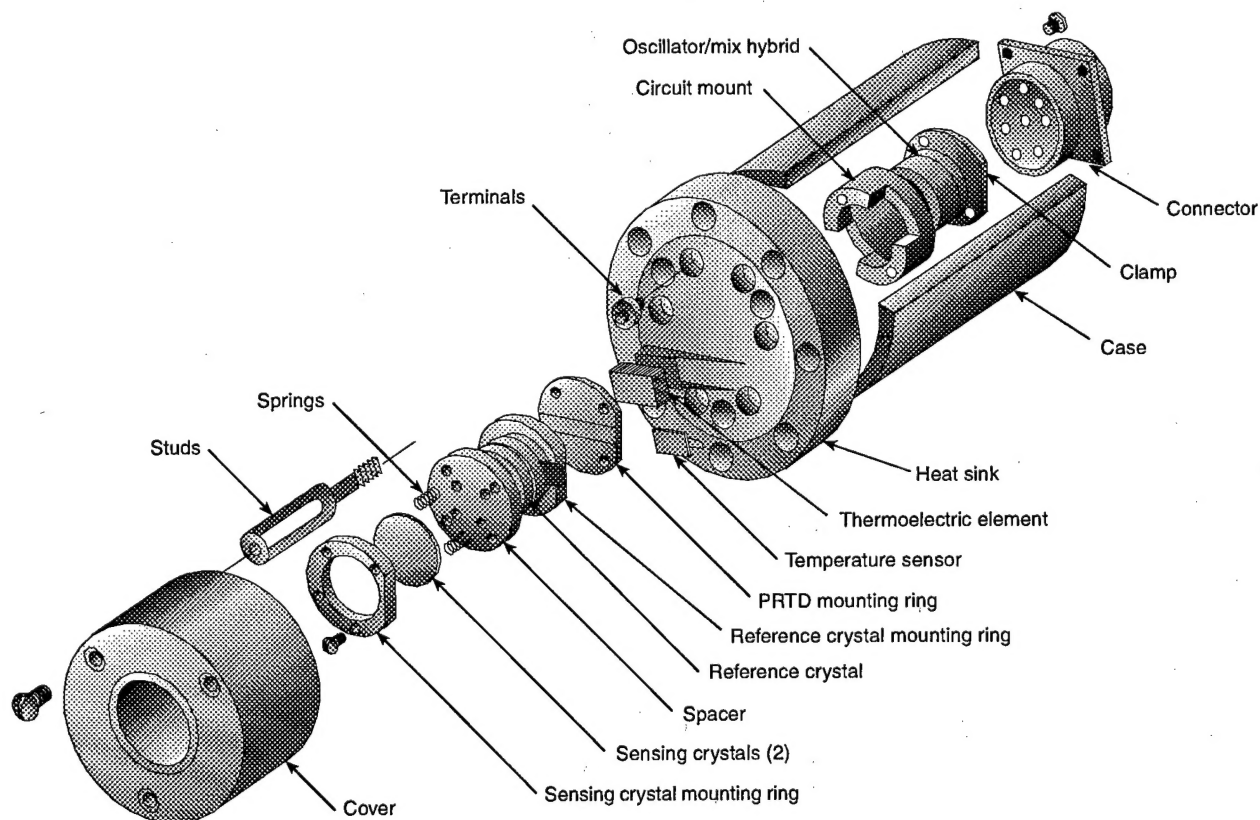


Figure 1. Configuration of a typical quartz crystal microbalance (PRTD = platinum resistance temperature device).

Temperature-to-frequency calibrations in vacuum were performed at the Arnold Engineering Development Center, Tullahoma, Tennessee,² for the MSX system, while similar calibrations were performed at APL for the SM-3 CSS sensors. The final beat frequency measurements (ΔF) were then converted into either percent obscuration (Obs) for the hydrocarbon-coated or "sticky" QCM serving as the particle sensor for the IR seeker, or into film thickness in micrometers for the uncoated QCM sensor. The QCM used in MSX monitored only film deposition because the sticky QCM was not well characterized at the time. A flow diagram of QCM data processing and analysis procedures is presented in Fig. 2.

For the wind tunnel tests,⁶ we chose the moderately clean particulate contamination level L of 650 from the Military Standard Product Cleanliness Levels and Contamination Control Program, MIL-STD-1246, as a starting point. This standard assumes a log-log² distribution of particle sizes, which is based on laboratory measurements of precision-cleaned hardware. One can then use the relationship for obscuration for spherical particles to the MIL-STD-1246 cleanliness level as⁷

$$\log(\text{Obs}) = 0.926 \log(L^2) - 7.245. \quad (1)$$

Using $L = 650$ in Eq. 1, one derives the percent obscuration of about 1%. One can now proceed to calculate the frequency output of the sensor for a 1% obscuration based on certain assumptions about particle size, shape, and density.

By assuming that the particles are spherical, one can calculate the total mass of particles captured on a perfectly accommodating sticky surface as

$$\Delta M = n\rho(4/3)\pi r^3, \quad (2)$$

where n is the number, ρ is the density, and r is the radius of the particles.

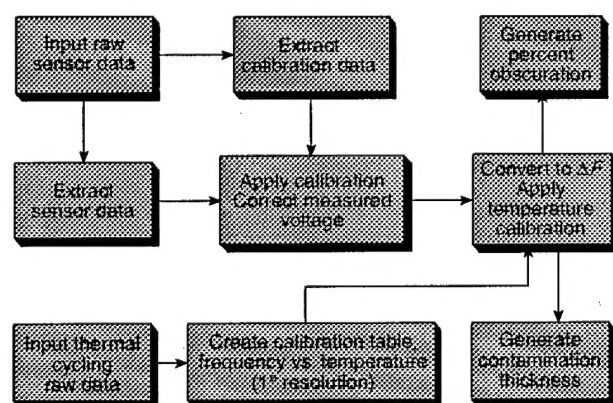


Figure 2. QCM sensor data processing and analysis procedures (ΔF = beat frequency measurements).

For a 1% obscuration,

$$A = 100a = 100n\pi r^2, \quad (3)$$

where A is the active surface area of the QCM, and a is the area obscured by the particle.

One then uses the basic operation equation³ of QCMs,

$$\Delta F = S_f(\Delta M/A), \quad (4)$$

where S_f is the sensitivity factor of the crystals, which is a function of the fundamental frequency, and ΔM is the mass loading on the outer oscillating crystal surface.

Substituting Eqs. 2 and 3 into Eq. 4, the beat frequency per 1% obscuration due to 10-mm particles is

$$\Delta F = S_f \rho (4/3) r (100)^{-1} = 1507 \text{ Hz}, \quad (5)$$

where S_f is experimentally measured³ as $2.260 \times 10^8 \text{ Hz g}^{-1} \text{ cm}^{-2}$, and ρ and r are assumed to be 1.0 g cm^{-3} and $5 \times 10^{-4} \text{ cm}$, respectively.

Thus, for this miniature QCM sensor, 1% obscuration is equivalent to a 1507-Hz frequency change. Since the response of the QCM sensor is directly proportional to the mass of contamination (Eq. 4), the maximum amount of contamination (and percent obscuration) depends on the full-scale range of the beat frequency designed into the signal conditioner. The CSS version used in the wind tunnel tests was designed to measure a maximum frequency range of 40,000 and can therefore measure 40,000/1507 or 26.5% obscuration. In reality, the hydrocarbon grease applied to the QCM accounts for about 5000 to 9000 Hz, so that the true range of the sticky QCM is 20 to 23% obscuration.

The conversion of the beat frequency ΔF measured by the QCM sensor into a film thickness deposited on the crystal is derived from the basic operation equation (Eq. 4). One can rearrange the units in terms of density and thickness as

$$\Delta F = S_f(\Delta M/A) = S_f \rho [\Delta t (10^4)], \quad (6)$$

where $S_f = 5.085 \times 10^8 \text{ Hz g}^{-1} \text{ cm}^{-2}$ for a 15-MHz crystal,³ ρ is the density of the film in g cm^{-3} , and Δt is the film thickness in micrometers. We used 15-MHz QCMs for the uncoated units because they were more sensitive. Rearranging Eq. 6 and using the CSS design with a maximum range of 40,000 Hz, the maximum film thickness that can be measured in the wind tunnel tests is

$$\Delta t = \Delta F [S_f \rho (10^4)]^{-1} = 0.787 \text{ } \mu\text{m}. \quad (7)$$

This film is thick enough to severely degrade the IR optics.^{1-3,8} With MSX and other NASA programs, a 0.1- μm (1000-Å) film of hydrocarbon contamination is considered very dirty for IR instruments. For UV instruments, hydrocarbon contamination as low as 10 Å is considered a problem, while the limits for visible instruments fall between the IR and UV regimes.⁸

SPACECRAFT APPLICATIONS

MSX (Fig. 3) was launched on 24 April 1996 into a 903-km, 99.4° orbit. MSX is a Ballistic Missile Defense Organization (BMDO) demonstration/validation program having both defense and civilian applications.^{4,9} With telescopes and imagers operating in the wavelength range from UV through IR, the spacecraft can identify and track ballistic missiles during mid-course flight, obtain data on test targets and space background phenomena, monitor in-flight contamination, and investigate the composition and dynamics of the Earth's atmosphere.

SPIRIT III (Fig. 4) is a major part of MSX; its sensor system components are cooled to temperatures ranging from 10 to 65 K. Contamination of the mirrors, windows, and detectors from condensed gases was of concern since the operational lifetime in space was projected to be about 18 months.⁴ Actually, it lasted only 10 months.¹ Contaminant mass deposition on the telescope primary mirror was monitored in real time through the use of a cryogenic QCM (CQCM) mounted adjacent to the mirror. The CQCM has been extremely useful for monitoring contaminant deposition during ground testing operations as well as in flight.¹⁰

In addition, four temperature-controlled QCMs (TQCMs) were mounted externally on the spacecraft at locations chosen to best characterize the real-time contamination phenomena as they occur around the external surfaces of MSX.^{1,4} Operating at -50°C, the TQCMs are too warm for deposition of water vapor but are cold enough to condense organic and silicone contaminants. Therefore, they provide a convenient method for monitoring these contaminants, which are the primary cause of long-term effects on thermal control coatings, power generation of the solar arrays, and decreased performance of optical telescopes.

Instrument Descriptions

CQCM

The primary mirror of SPIRIT III (Fig. 4) is of most concern to MSX since it faces the entrance aperture through which most of the contaminants pass. Located adjacent to the primary mirror and operating at the same temperature (about 20 K) as the cryogenically

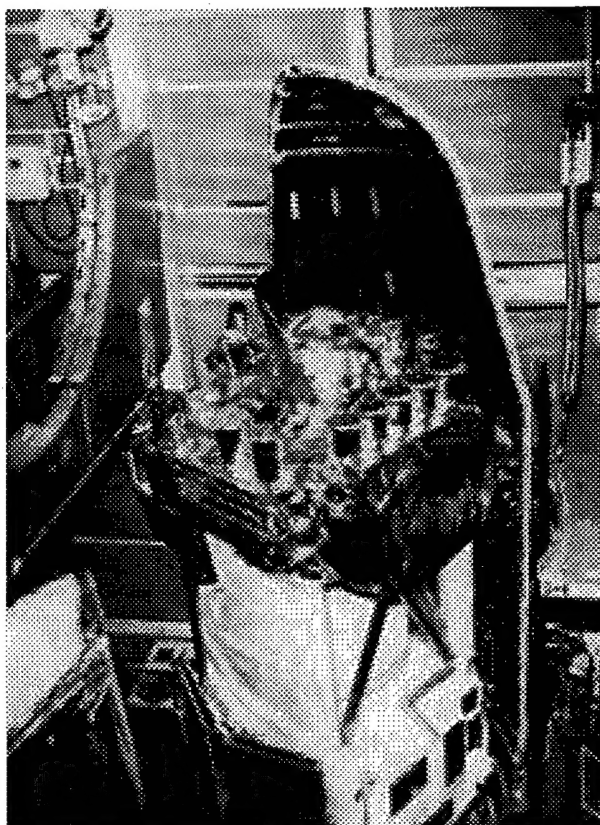


Figure 3. MSX partially covered with a shroud just before launch.

cooled optics, it is the only contamination monitoring sensor of the SPIRIT III instrument.⁹ The CQCM selected was the smallest QCM available at the time that could operate cryogenically and had the capability for thermogravimetric analysis (TGA). During TGA, the sample is heated at a uniform rate while simultaneously measuring mass and temperature. The temperature at which the substance evaporates is then used to identify its composition. The Mark 16 model from QCM Research, Laguna Beach, California (Fig. 5), was found to be compatible with the size, power, and technical requirements.¹¹

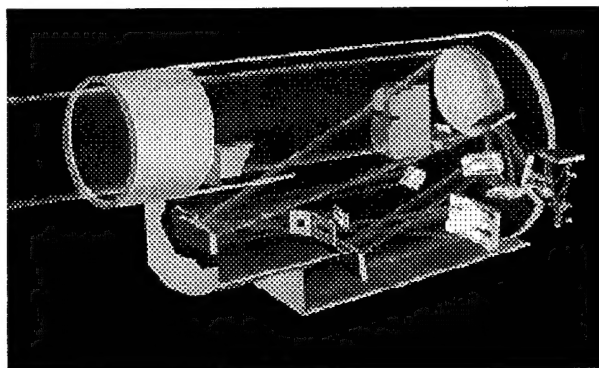


Figure 4. Drawing of the SPIRIT III telescope with the cryogenic QCM (CQCM) mounted next to the primary mirror.

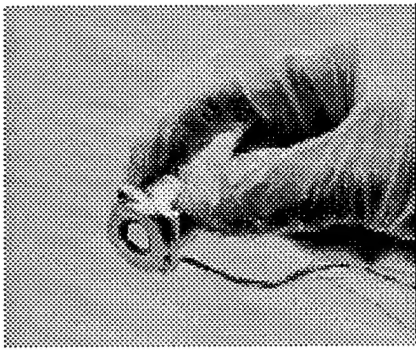


Figure 5. The Mark 16 CQCM used in MSX.

The CQCM on MSX is adjacent to and thermally coupled to the cryogenically cooled primary mirror of the SPIRIT III telescope. It was used to monitor deposition of contaminants on the interior optics and, with associated optical data, to determine the degradation in mirror performance.

After installation in the SPIRIT III telescope, the CQCM was a valuable tool for monitoring the mirror status during cryogenic testing at Utah State University's Space Dynamics Laboratory, thermal vacuum testing at the NASA Goddard Space Flight Center, and preflight measurements at the launch site.¹⁰

TQCMs

The MSX TQCMs¹² are modified Mark 10 units, also built by QCM Research, and were designed to operate at temperatures between -70 to $+70^{\circ}\text{C}$ (Fig. 6). Use of QCMs from the same vendor allowed for a simplified design of the controller and signal conditioner. The temperatures are controlled by a Peltier cooler/heater unit that is built into these Mark 10 units. When in the "park" mode, the $+x$ face of the spacecraft points away from Earth and is perpendicular (90°) to the Sun direction to minimize thermal heating. At this orientation, the $-y$ face is also perpendicular (90°) to the Sun direction to give maximum solar array output. Thus, TQCM 1 is pointed with components in the $(-x, y, z)$ directions, TQCM 2 points in the $+z$ direction, TQCM 3 has $(y, -z)$ components, and TQCM 4 has $(x, -y, z)$ components. The TQCM covers limit their field of view to a right cone with an approximately 64° half-angle. TQCMs 1 and 2 both have fields of view that contain a considerable area of the solar panels, although TQCM 2 generally is exposed to the spacecraft's direction of travel during orbit. TQCM 3 looks in the wake direction, where minimal contamination is expected. TQCM 4 is mounted on the $+x$ face of the spacecraft and thus provides the deposition rate on the surfaces where all of the optical instruments are pointing. Because the spacecraft was predicted to cool to

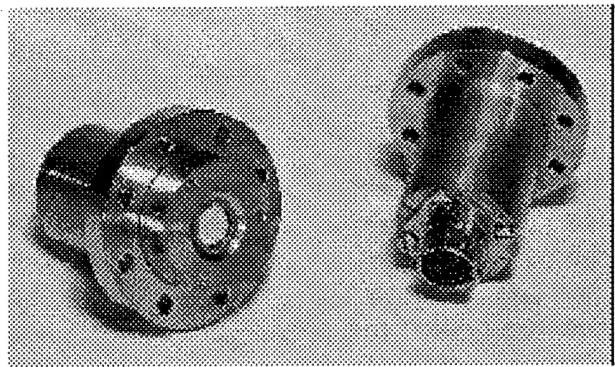


Figure 6. The Mark 10 TQCMs in MSX.

temperatures on the order of -20°C , the deposition levels measured by the TQCMs at -50°C represent a "worst case" condition for the UV and visible instruments of the UVISI (Ultraviolet and Visible Spectrographs and Imagers) and the SBV (Space Based Visible) telescope. Unlike the CQCM, the TQCMs are routinely exposed to solar radiation since they are mounted externally to the spacecraft. The radiation causes a momentary decrease in TQCM output frequency depending on the solar flux.

MSX Results

The mass accretion recorded by the QCMs during the first 10 months in orbit is shown in Fig. 7. As expected, the mass accretion inside the SPIRIT III near the CQCM was rapid because of the extremely low temperature of the sensor surface, which allowed condensation of gases like argon, oxygen, and nitrogen as well as water vapor. But after about 60 days in orbit, the CQCM mass accretion was minimal. The TQCMs, however, appear to have a more gradual mass accumulation, except for TQCM 3, which is the wake-facing sensor and is expected to be on the clean side.

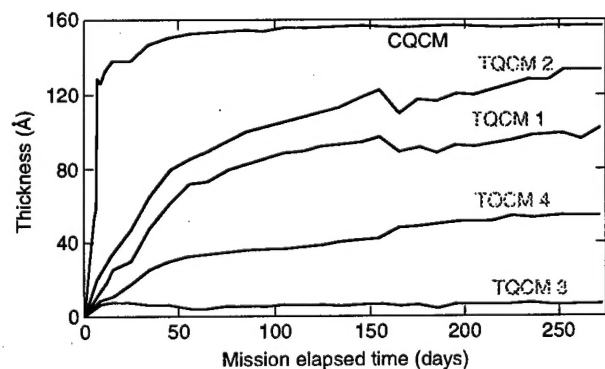


Figure 7. Accreted contaminant film thickness versus mission elapsed time for the CQCM and all four TQCMs.

In addition to monitoring mass accretion on surfaces, these QCM sensors can be used to identify the molecular species of the contaminants using TGA. In the CQCM TGA mode, the condensed species on the QCM can be identified from their respective sublimation temperatures.

From a prelaunch model prediction, the heaviest contamination was expected to occur either on the ram-facing TQCM 2 or the solar array-facing TQCM 1.¹³ Because TQCM 2 also has some view of the solar array, it might exceed TQCM 1 contamination depending on relative contributions from both sources. TQCM 3 was expected to be the least contaminated, while TQCM 4 was expected to be somewhere in between TQCMs 2 and 3. Figure 7 shows that this is indeed the case, thus validating the MSX contamination model.¹³

After the SPIRIT III dewar had warmed to a temperature where the focal plane arrays were no longer usable, a series of warm-up experiments, called the SPIRIT III End of Cryogenic Operations Tests (SECOT), was undertaken to warm the baffles up to evaporate all of the H₂O and CO₂ that had been condensed during the cryogenic period. The spacecraft was positioned to allow part of the Sun radiation to enter through the front aperture. The CQCM was located at the back end of the telescope nearest the cryogen dewar and, therefore, remained sufficiently cold (<100 K) to condense water and higher molecular weight gases. Warming the baffles above 160 K by a series of solar radiation exposures essentially cleaned them, even though the temperatures were later increased to above 200 K.

During this heating series, the CQCM recorded the condensed mass during each of the baffle heating pulses. After the deposition phase had been completed, a TGA of the mass condensed was performed, and the results can be seen in Fig. 8. The contaminant film thickness is displayed versus CQCM crystal temperature. Nearly all of the approximately 200-Å-thick film that had condensed during the heating pulses evaporated between 150 and 160 K, indicating that essentially all of the film was water. It also indicates that the cryogenically cooled baffles were very effective in protecting the primary mirror from water deposition, since no trace of water had been seen during previous CQCM TGAs.

Since the CQCM condensed only a small portion of the gas that had evaporated during SECOT, it is desirable to know how much water had been originally condensed on the baffles before the warm-ups. The SECOT heating experiments and SPIRIT III baffle temperatures are being modeled to determine the total water evaporated. This effort is ongoing, and the results will be reported in a later article. The source of the H₂O is thought to be outgassing from surrounding multilayer

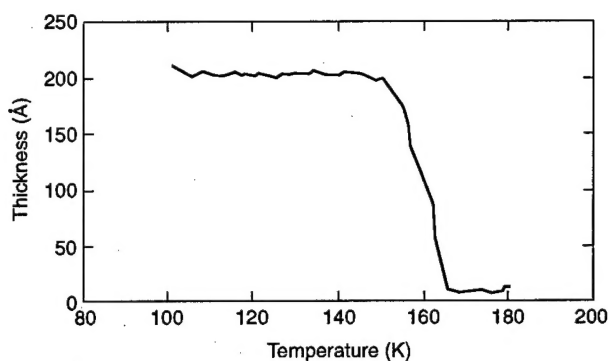


Figure 8. Results of the CQCM thermogravimetric analysis performed after SPIRIT III baffle heating during End of Cryogenic Operations Tests showing that the contaminant is water ice, which sublimates at 150 K in high vacuum.

insulation blankets and outgassing products from external materials on the spacecraft that entered through the SPIRIT III entrance aperture.

Spacecraft Application Summary

The QCMs flown with MSX have proven useful for monitoring the on-orbit contaminant mass accretion on the SPIRIT III cryogenic telescope primary mirror and on the satellite's external surfaces. After 272 days in orbit, the CQCM (and primary mirror) accumulated 155 Å of condensate on the 20 K surfaces. Most of this condensate was due to the argon condensed during the SPIRIT III cover release. The cover was cooled with liquid argon before release to minimize cryogen loss of the SPIRIT III cryostat. Essentially all of the condensate has been argon and oxygen, less than 1% being water as determined by TGAs before and after cover release.

Ground tests on the optical effects of condensed films on cryogenic mirrors at 20 K indicate that the 155-Å film has had negligible effect on the mirror scatter and reflectance. The four TQCMs mounted on external surfaces have been operated at temperatures of about -50°C and have shown accumulations between 7 and 134 Å, depending on the location of the TQCM. The TQCMs with the solar panels in their field of view have shown the largest deposition rates. They are continuing to accumulate mass, and the long-term trends established for MSX will be extremely valuable for future satellite systems.

Warming of the SPIRIT III baffles at the end of the cryogenic lifetime resulted in the deposition of approximately 200 Å of H₂O on the CQCM. The source of this water is believed to be outgassing from the multilayer insulation within SPIRIT III and from external surfaces.

MISSILE APPLICATIONS

As mentioned previously, our experience in the use of QCMs in MSX uniquely qualified APL to fabricate and support the upcoming flight tests with a CSS to meet the SM-3 program requirements. Figure 9 shows the locations of two of the four TQCM sensors in the missile area. Because the flight tests had not yet been launched at the time of this writing, only the rationale for the design of the APL CSS and the early results of the preflight wind tunnel tests are reported here.

To reduce risk before the missile flight tests, the candidate nose cones were tested in the APL wind tunnel facility to assess their performance with respect to the temperature and pressure environment. When contamination appeared to exist,⁵ a second series of wind tunnel tests was designed that included the CSS as one of several sensors. The purpose of the second series was twofold: to assess the reduction of contamination due to fabrication changes of the nose cone and to determine the effect of the nose cone on seeker performance during a simulated flight. Figure 10 shows the SM-3 nose cone test hardware and locations of the QCMs during the wind tunnel tests.

Rationale

The only real-time contamination sensor that fit into the volume available within the nose cone was a miniature QCM called the Mark 21 (Fig. 11), also manufactured by QCM Research.¹⁴ Although the Mark 21 QCMs are as sensitive as the Mark 10 and 16 units used in MSX, they weigh only 4.7 g and have a volume of about 10.8 cm³. Because of the tight schedule, we chose to minimize design changes to the QCM sensors and to qualify them by selection through incoming acceptance inspection and environmental testing. The overall flight qualification of this sensor was accomplished through a variety of environmental tests as specified in the ICD.⁶ Several techniques were implemented to reduce development time:

- The power section and digital/analog conversion circuitry were designed around commercial off-the-shelf components.
- A field-programmable gate array was used to provide greater flexibility, since the ICD was in the process of being defined.

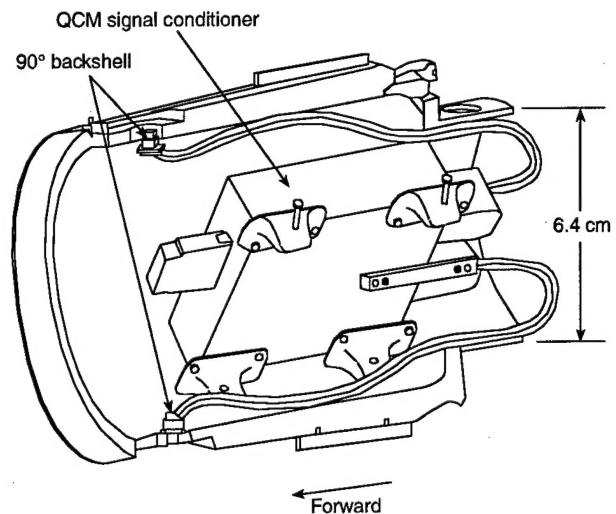


Figure 9. Contamination Sensor System configuration in the SM-3 thruster area.

- The QCM analog interface section was based on the commercial design to minimize potential interface problems.
- The vendor's cables were to be used as delivered since they were fabricated for a clean space environment and low-noise operation.

Since QCMs respond to mass accretion on the crystal surface, these units can also be used to measure solid particles landing and depositing on the crystal,¹⁵ but the

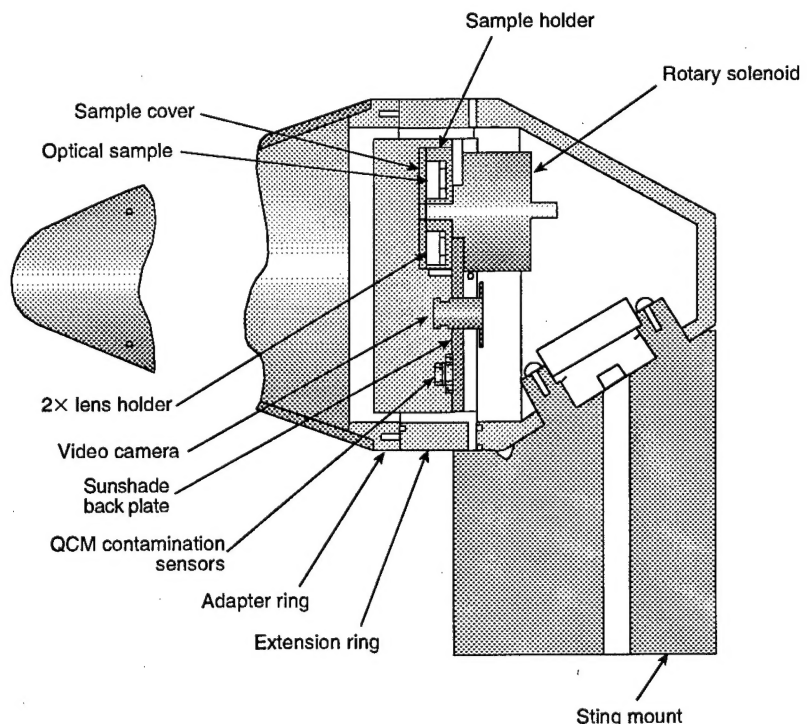


Figure 10. SM-3 nose cone test hardware showing the location of the QCMs during the wind tunnel tests.

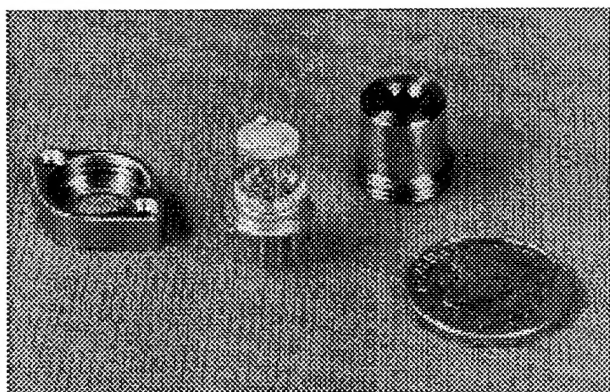


Figure 11. The Mark 21 miniature QCM.

adhesion of the particles on a clean silicon surface is not well characterized and depends on the particle size and impact velocity.¹⁶ A particle of more than 3- μ m diameter and with an impact velocity of more than 3 m/s will rebound and not adhere to the silicon surface.¹⁷ However, when the exposed QCM surface has been coated with a sticky material such as Apiezon grease, particles with diameters up to 15 μ m and impact velocities of >0.37 m/s have been found to adhere to the sticky surface.^{15,17} (Apiezon grease is a commonly used sealing material in high vacuum applications. It is the sticky layer used to provide the particles with the necessary coupling to the surface of the QCM crystal so that its mass can be measured as a change in the beat frequency.) Even though QCMs have also been used to measure particles during spaceflight,³ the most recent application of sticky QCMs to measure particles in space was with the Mars Rover.^{18,19}

Because of this history, the sticky QCM particulate sensor was added to the more traditional QCMs (i.e., as a molecular condensable film sensor) in the missile flight tests and the wind tunnel tests preceding the flight tests. Although other particulate monitors are known to detect slow-moving particles,²⁰ none could be found that were compatible with the space and power requirements of the SM-3 kinetic warhead.⁶

Design

In addition to the successful spaceflight history of QCMs,^{1-3,18-20} another advantage of using them for both film and particulate contamination sensing is the use of a common signal-conditioning module. In the SM-3 flight test application, APL designed, fabricated, and flight qualified a four-channel signal conditioning module to be used with both molecular film and particle deposit sensors because both operate on the same physical and electronic principles (Fig. 9).

The missile telemetry system is analog-based, and the CSS was designed to match the interface of the pressure channels, 0 to 5 V. The CSS provides a current

source for the platinum resistance temperature detectors (PRTDs) and a voltage source for the oscillators to the QCMs. Because knowledge of the crystal temperature is essential to monitoring condensation and evaporation of materials on the QCM, all QCMs also measure temperatures as well as beat frequencies. Temperature is monitored from -28 to $100 \pm 0.06^\circ\text{C}$. The beat frequency of the QCM is counted and converted to an analog level via the digital-to-analog converter. The QCMs in the mirror optics are more sensitive from 0 to 40640 ± 20 Hz, which (from Eqs. 5 and 7) theoretically corresponds to 0 to $26.96\% \pm 0.01\%$ obscuration on coated sensors or 0 to 0.799 ± 0.001 μ m on uncoated sensors. The system requires 4200 mW, of which 560 mW is needed for the four QCM sensors. The electronic block diagram of the CSS showing all QCM sensors and PRTDs is shown in Fig. 12.

The units successfully passed vibration and shock tests as well as thermal cycling. To qualify the design by similarity, one unit also successfully underwent travel vibration, humidity, corona, and electromagnetic interference susceptibility testing. The flight and flight spare were installed in the SM-3 missile and have successfully passed environmental testing at the prime contractor's facilities. At the time of this writing, they are in various stages of preparation for launch. Results of these flight tests will be reported in a follow-up article.

Results

In the wind tunnel tests, two QCMs, one to detect particulate and the other to detect molecular deposition, were mounted on the sample holder shown in Fig. 10. Even though the CSS signal conditioner module was designed to interface with four QCM sensors, the wind tunnel tests required the use of only two. Note that the holder contained two optical witness mirrors, which have a solenoid-operated shutter to open and close them at the start and end of the flight portion of the wind tunnel tests. There was also a video camera, which earlier provided evidence of smoke contamination.²¹ A series of four tests (FEF 165 through FEF 168) instrumented with QCMs was performed in the wind tunnel. The test nomenclatures are those assigned by APL's AATDL and are maintained for correctness in this article. Since the last two tests (FEF 167 and 168) duplicated the first two,²² only the former (FEF 165 and FEF 166) are reported here.

Test FEF 165

FEF 165 was a test of the baseline nose cone configuration with a seeker cover installed (see Refs. 22 and 23 for a detailed description). The nose cone was tested for the entire 150-s flight duration, but the QCM sensors recorded data (temperature and frequency) about 30 s before and about 70 s after the flight portion

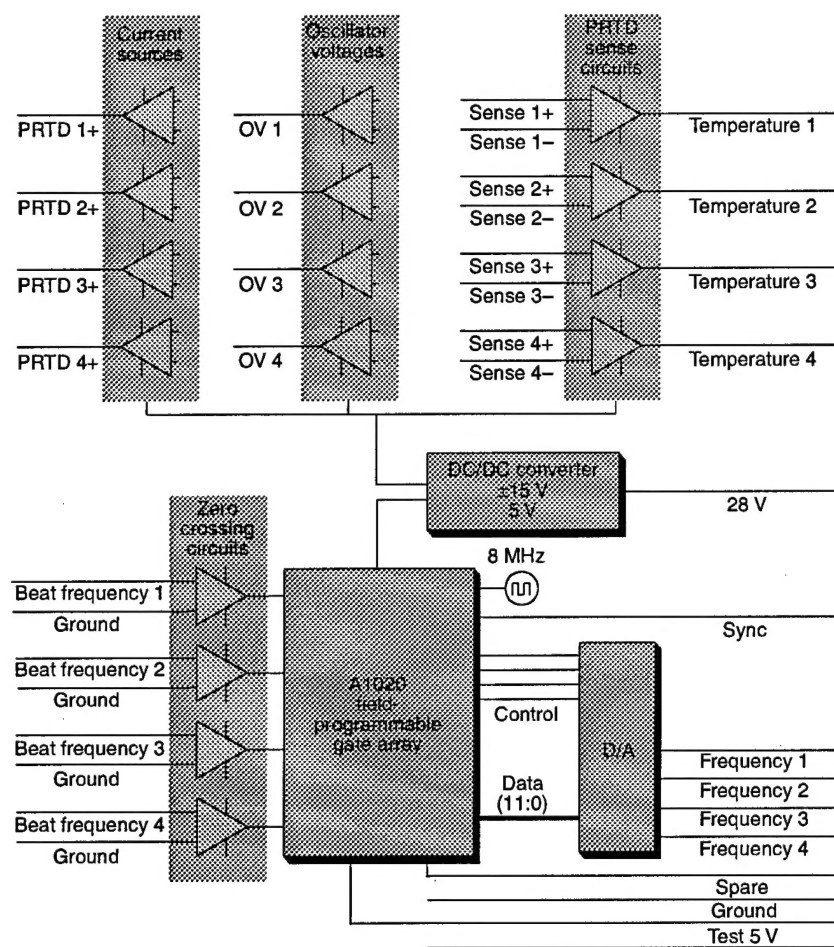


Figure 12. Electronic block diagram of the CSS showing all QCM sensors, PRTDs, oscillator voltages (OV), and ground.

of the test. The QCM sensors did not detect any significant contamination from either particulate or molecular deposition before or during flight, but did detect very large contamination effects after flight due to facility shutdown procedures (Figs. 13 and 14).

Figure 13 shows the percent obscuration measured by coated QCM 1498, with saturation several seconds after shutdown. The saturation occurred at about 22% obscuration because of a "zero" correction applied to the data of about 4% due to residual loading of the QCM sensor before the test began. This residual was attributable to the thin film of Apiezon grease intentionally applied on the crystal as the sticky layer and probably also from a small amount of contamination during handling and testing.

The uncoated QCM 1998 for testing molecular film deposition (Fig. 14) measured no significant contamination until after the flight was completed and also showed a saturation peak about 20 s after shutdown, slightly later than the saturation of the particulate QCM 1498. It also appeared to have lost its initial deposits at about 220 s, only to regain them after about 10 to 20 s. Because this QCM did not have a sticky

surface, this loss seemed to occur where the deposited material either evaporated or fell off the surface, only to get additional deposition later. The phenomenon was later verified when the sensors were examined in the scanning electron microscope (SEM) as part of a posttest analysis.

A posttest SEM image (Fig. 15a) shows the particles embedded on the surface of QCM 1998. The molecular film deposits are the dark areas. Note that there are still light circular areas that proved to be clean gold surfaces indicative of the original QCM crystal, which is gold-coated. Elemental analysis by energy-dispersive X ray (EDX) in the SEM showed only a high carbon and slight oxygen composition of the dark areas and dark deposits,²² a clear indication of hydrocarbon contamination. The light areas showed only gold and the underlying silicon and oxygen due to quartz.²² Figure 15a, which shows these previously covered areas, supports the results shown in Fig. 14, i.e., particles may have been deposited and then fallen off the surface of the QCM. On another area of the QCM crystal, a much larger,

serpentine deposit was observed (Fig. 15b). The shape suggests that the deposits were sprayed on the surface as liquid droplets, which coagulated into a lengthier solid. The serpentine deposit is about 200 μm long.

Our initial conclusion regarding FEF 165 is that the seeker cover provided a significant contamination barrier for seeker optics since the QCMs detected almost no contamination during the trajectory portion of the wind tunnel test. After the test, the contamination was substantial, with both particulate and molecular film due to the nose cone's continued outgassing as well as gas leakage from the outside after the wind tunnel was shut down.

Test FEF 166

This was a test of the baseline nose cone configuration with a radiation shield but no seeker cover. Figure 16 shows the contamination buildup of particles on the coated QCM 2698 during this test. It indicated that particles started to deposit on the sensor at about 50 s into the flight, went full-scale to about 22% obscuration (corrected for a residual loading of about 4%)

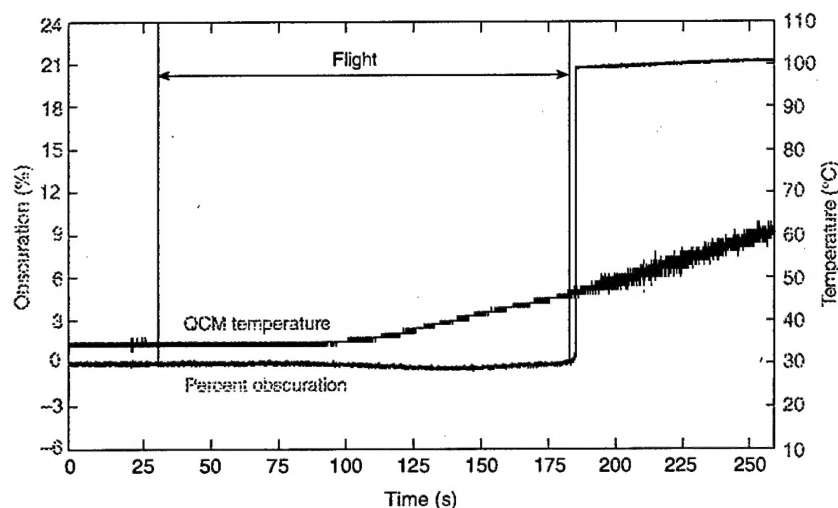


Figure 13. FEF 165 QCM 1498 (coated) percent obscuration results from the wind tunnel test with timing errors corrected.

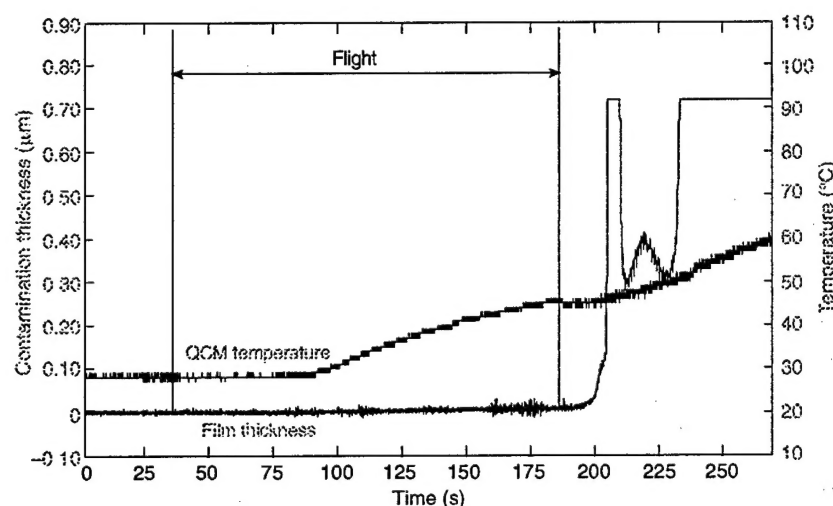


Figure 14. FEF 165 QCM 1998 (uncoated) film deposition during the wind tunnel test.

at about 70 s, and stayed at full-scale for the duration of the measurement.

QCM 4297 (Fig. 17) showed a very small amount of contamination on the uncoated crystal surface about 50 s into the flight, seems to have lost the contamination around 90 s, and then acquired about 0.03 μm of deposit until the flight ended. As was true with the previous test FEF 165, the temperature inside the nose cone continued to increase so that at about 10 s after shutoff, the molecular deposits on QCM 4297 went full-scale to about 0.7 μm and remained saturated to the end of the test.²²

Posttest SEM images of the film deposition and the particles on the sticky surface (Figs. 18 and 19, respectively) clearly show the amount of contamination. The particle shapes appear irregular and large in Fig. 19 because it is difficult to distinguish the grease "islands" from the actual particles trapped within

them. Elemental analysis by EDX of the particles and molecular film confirmed that they were carbonaceous, largely carbon and oxygen.²²

In test FEF 166, the appearance and elemental composition of the contamination from the QCM sensors and witness mirrors appear to be due to hydrocarbons from the nose cone materials. Since this test is almost the same as test FEF 165, except for the absence of a seeker cover, one must conclude that the seeker cover has a major beneficial effect on the reduction of seeker contamination.

Missile Application Summary

Wind tunnel tests conducted in the Cell 2 Mach 6 facility at APL have verified the ability of QCMs to measure real-time contamination in flight conditions. They have also verified that the seeker cover offers good protection against contamination of the seeker optics. The various modifications tested during this series of wind tunnel tests led the prime contractor to further reduce the potential contamination in flight to levels required for a successful mission.

NEXT-GENERATION QCM SENSOR

The next-generation QCM sensor design will provide a common system for both space and missile applications. Neither the SPIRIT III nor the SM-3 miniature QCMs had combined active temperature-controller and TGA capability because of size and power constraints. What is required is a QCM sensor of about the same size as the Mark 21, but having the capabilities of the larger Mark 10 TQCMs. At APL's request, and based on our experience with the Mark 21 units, QCM Research³ implemented several improvements, e.g.,

- Kapton isolation washers over the connectors
- Longer mounting leads
- Improved machining tolerances
- Pin identification
- Staking of the internal electronics carrier and cover

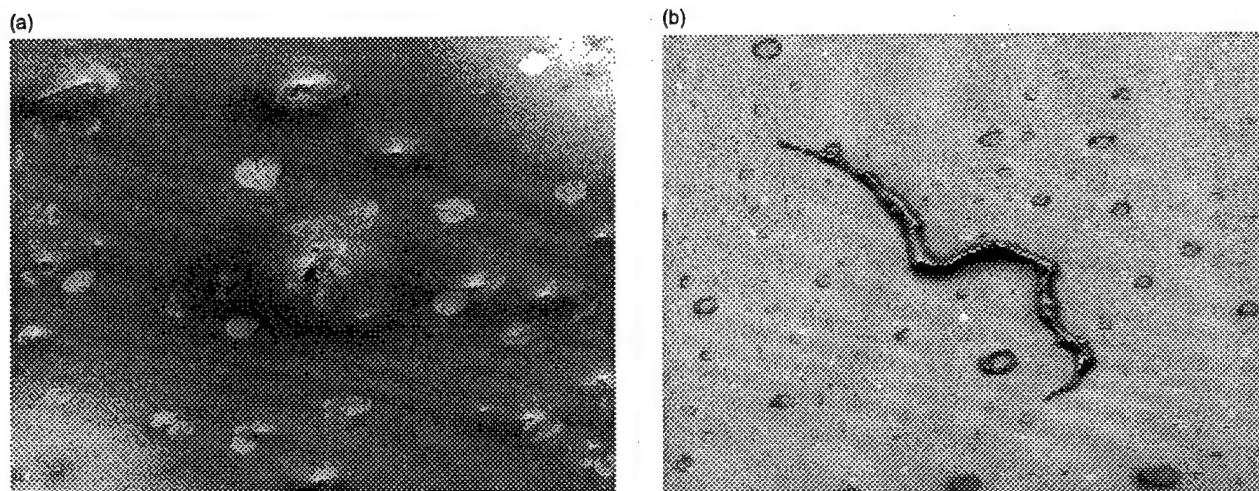


Figure 15. FEF 165 QCM 1998 SEM photograph (a) at 1000 \times magnification showing particles as well as clean areas previously protected from contamination, and (b) at 500 \times magnification showing a serpentine deposit.

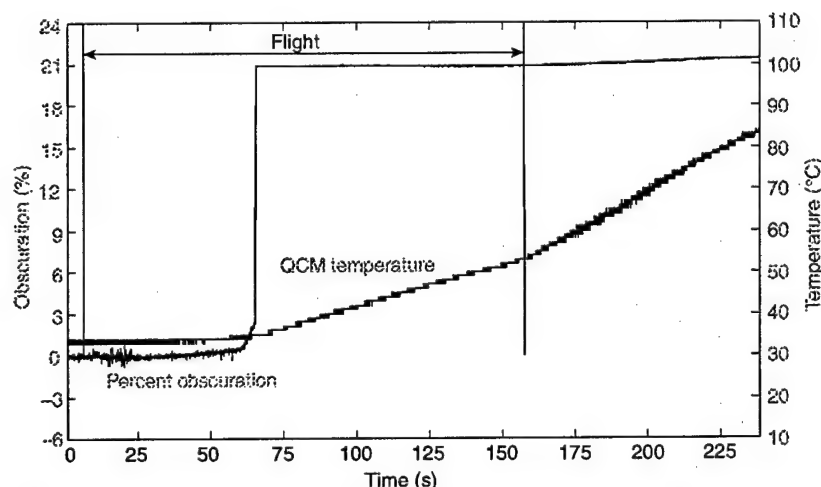


Figure 16. FEF 166 coated QCM 2698 showing full-scale readings of percent obscuration at about 70 s after the start of the wind tunnel test.

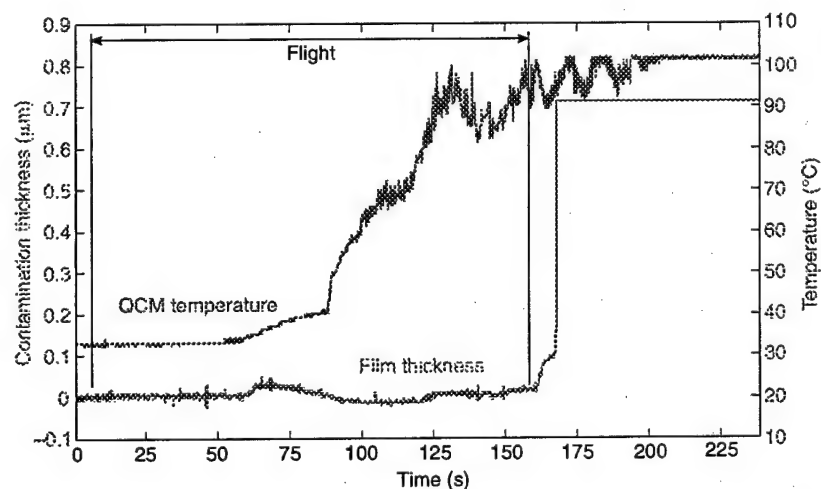


Figure 17. FEF 166 QCM 4297 showing small film deposition during trajectory until after the test.

In addition, we wanted to install a miniature thermoelectric cooler (TEC) inside the QCM to give it a TGA function. A TEC is based on the Peltier effect in which cooling/heating occur when electrical current passes through two conductors. Upon application of a voltage across two dissimilar materials, a temperature difference is created and heat moves from one end to the other end. A Marlow MI1020T TEC was installed into a redesigned Mark 21 unit (now called Mark 24 by QCM Research) which can theoretically lower the crystal temperature 67°C below ambient in vacuum. The MI1020 is made of bismuth telluride and draws 1.8 A at 0.8 V. The TEC is also used for heating the QCM crystals for TGA. As Fig. 8 illustrates, the contaminants sublime, evaporate, or decompose in vacuum, with the corresponding decrease in the beat frequency. From the resulting frequency reduction versus time and temperature, the mass flux can be calculated and the vapor pressures can be determined. Either the vapor pressure or the vaporization temperature of the unknown species can then be used to identify its composition. A laboratory TGA calibration of several

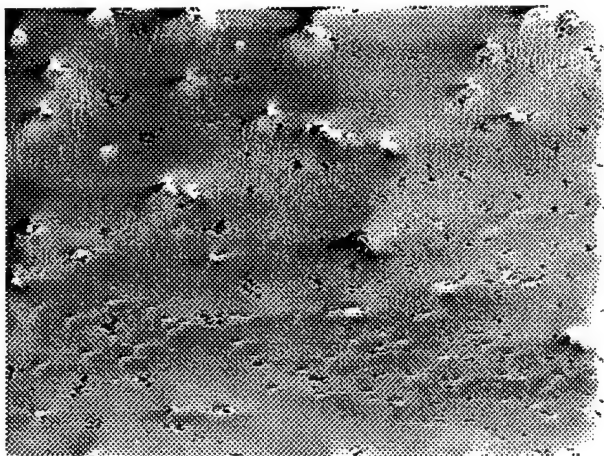


Figure 18. FEF QCM 4297 SEM image of the uncoated crystal showing a "hilly" appearance for the molecular film.

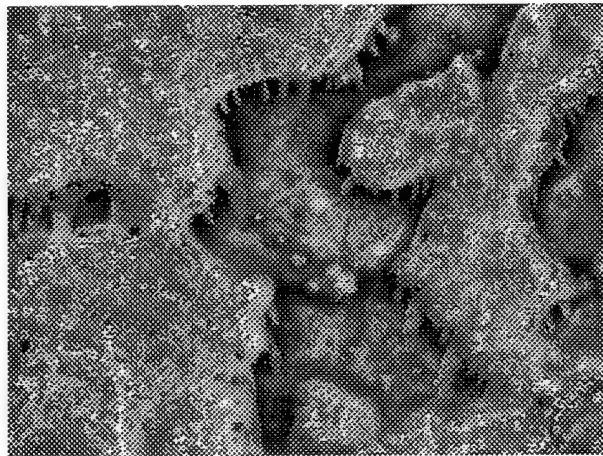


Figure 19. SEM image of QCM 2698 showing particles trapped by the Apiezon grease.

expected contaminants would make TGA even more specific.

Design

The design for a combined space and missile QCM sensor—a cooperative venture with QCM Research, which is providing a newly designed Mark 24—takes into account new technologies and APL's advanced

manufacturing skills. It will provide substantially improved features, a 10-fold reduction in space over the original CSS design and a 100-fold reduction over the commercial controller. The new design is based on an innovative part from Analog Devices, the ADuC812. It combines an 8051 microprocessor, two 12-bit digital-to-analog converters, one self-calibrating true 12-bit analog-to-digital converter, an 8-channel single-ended multiplexer, and several other features in a 52-pin

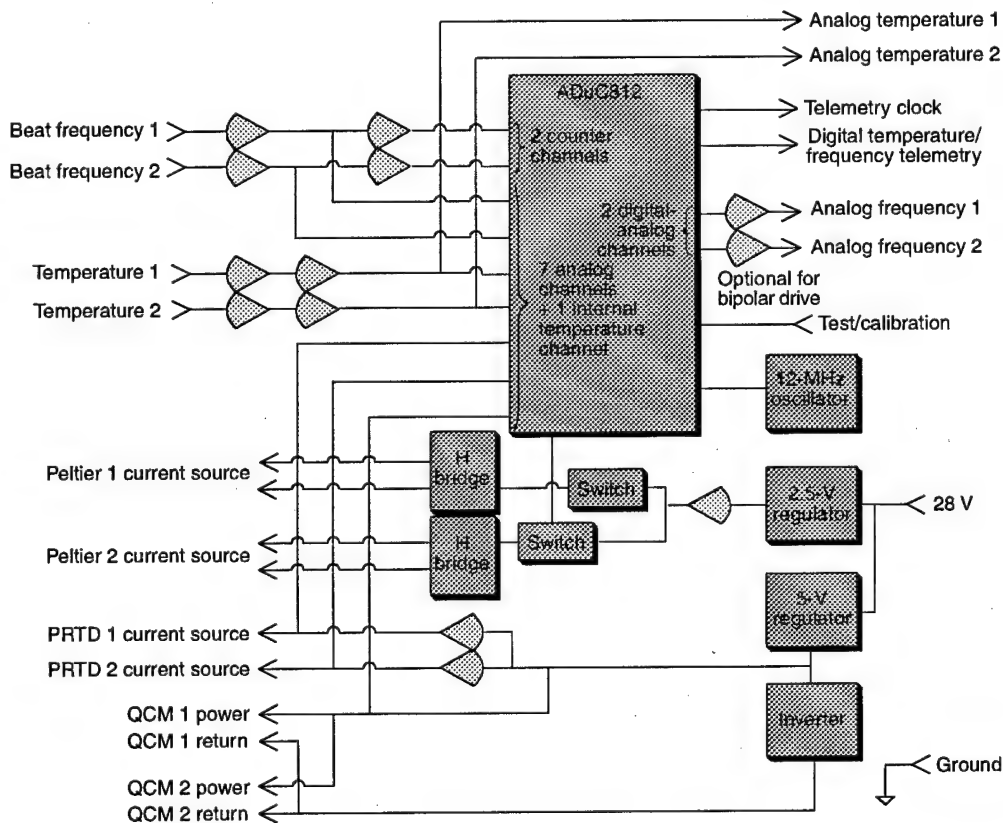


Figure 20. Electrical schematic of the new design for both space and missile applications showing all PRTDs and oscillators.

plastic quad flat package. Most of the operational amplifiers and voltage comparators used in the design are housed in a new SC70-5 package, which only requires 4 mm² of board space. The power systems consist of a 5-V DC-to-DC converter, a +5- to -5-V inverter, and a 2.5-V DC-to-DC converter. The QCMs operate from the ± 5 -V supplies, while the 2.5-V supply is used by the TECs.

Preliminary Results

Parts selection and acquisition are complete, and a breadboard design has begun. The redesigned QCM and controller have completed mechanical development and are in electrical fabrication. Newly designed miniature connectors called Nanonics²⁴ will also be used. Initial sizing suggests that the controller will be a 3.8-cm-dia. \times 5-cm-long cylindrical housing volumetric target. The functionality and flexibility of the design will be much greater than the current CSS design. The power requirements for the controller and QCMs will be below 0.25 W, while the TECs will draw nearly 4 W. The schematic of this new system is shown in Fig. 20.

CONCLUSION

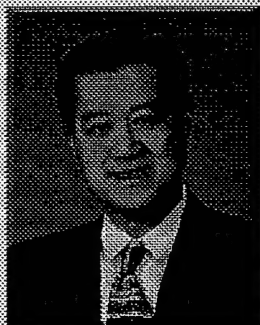
Miniature QCM sensor systems have been successfully designed, fabricated, and flight-qualified at APL for space and missile applications. A more compact system has been designed and is being fabricated for both applications, and should be ready for flight qualification very soon. Such a miniature QCM sensor will decrease size and power requirements, and the use of commercial off-the-shelf components will also decrease cost. Several potential sponsors have already expressed interest in this system.

REFERENCES

- ¹Uy, O. M., Benson, R. C., Erlandson, R. E., Boies, M. T., Silver, D. M., et al., "MSX Contamination Research: Measurement of Gaseous and Particulate Environments During the Cryogen Phase," *J. Spacecr. Rockets* 35(2), 170-176 (1998).
- ²Wood, B. E., Hall, D. F., Lesho, J. C., Uy, O. M., and Dyer, J. S., "Midcourse Space Experiment Satellite Flight Measurements of Contaminants on Quartz Crystal Microbalances," *J. Spacecr. Rockets* 35(4), 533-538 (1998).
- ³Wallace, D., and Wallace, S., *History of Quartz Crystal Microbalance Development for Space*, QCM Research, Laguna Beach, CA, available at <http://www.qcmresearch.com/flthist.htm> (accessed 29 Jan 1999).
- ⁴Mill, J. D., O'Neil, R., Price, S., Romick, G., Uy, O. M., et al., "Midcourse Space Experiment: An Introduction to the Spacecraft, Instruments and Scientific Objectives," *J. Spacecr. Rockets* 31(5), 900-907 (1994).

- ⁵Linevsky, M. J., *Reflectance and Transmittance Spectra of Exposed Mirror and ZnS Samples*, A1F (5) 98-U-047, JHU/APL, Laurel, MD (29 May 1998).
- ⁶SM-3 CTV-1A Contamination Sensor System Interface Control Document, DWG No. 6025-1050, JHU/APL, Laurel, MD (1 Jun 1998).
- ⁷Ma, P. T., Fong, M. C., and Lee, A. L., "Surface Particle Obscuration and BRDF Predictions," in *Proc. Scatter from Optical Components Conf.*, SPIE 1165, 381-391 (1989).
- ⁸Tribble, A. C., Boyadjian, B., Davis, J., Haffner, J., and McCullough, E., *Contamination Control Engineering Design Guidelines for the Aerospace Community*, NASA Contractor Report 4740 from Rockwell Int. Corp. (May 1996).
- ⁹Wood, B. E., Bertrand, W. T., Hall, D. F., Lesho, J. C., Uy, O. M., and Dyer, J. S., *MSX Satellite Flight Measurements of Contaminant Deposition on a QCM and on TQCMs*, AIAA Paper No. 97-0841, 35th Aerospace Sciences Meeting and Exhibit, Reno, NV (6-10 Jan 1997).
- ¹⁰Dyer, J. S., Mikesell, R., Perry, R., Mikesell, T., and Guregian, J. J., "Contamination Measurements During Development and Testing of the SPIRIT III Cryogenic Infrared Telescope," *Proc. SPIE* 2261, 239-253 (1994).
- ¹¹Wallace, D., and Wallace, S., *The MK16TM, MK17TM and MK18TM Cryogenic Quartz Crystal Microbalances*, QCM Research, Laguna Beach, CA, available at <http://www.qcmresearch.com/qcm.htm> (accessed 1 Feb 1999).
- ¹²Wallace, D., and Wallace, S., *MK10 TQCMTM Sensor, The Industry Standard*, QCM Research, Laguna Beach, CA, available at <http://www.qcmresearch.com/mk10.htm> (accessed 1 Feb 1999).
- ¹³Silver, D. M., *Midcourse Space Experiment Modeling*, AIAA 96-0223, 34th Aerospace Sciences Meeting and Exhibit, Reno, NV (6-10 Jan 1996).
- ¹⁴Wallace, D., and Wallace, S., *Mark 21 Miniature QCMTM*, QCM Research, Laguna Beach, CA, available at <http://www.qcmresearch.com/mk21.htm> (accessed 1 Feb 1999).
- ¹⁵Woods, D. C., "Measurement of Particulate Aerosol Mass Concentration Using a Piezoelectric Crystal Microbalance," in *Aerosol Measurements*, D. A. Lundgren, F. S. Harris, W. H. Marlow, et al., (eds.), University Presses of Florida, Gainesville, pp. 119-130 (1979).
- ¹⁶John, W., "Particle-Surface Interactions Charge Transfer, Energy Loss, Resuspension and Deagglomeration," *Aerosol Sci. Tech.* 23(1), 101-102 (1 Jul 1995).
- ¹⁷Caylor, M. J., Dunn, P. F., and Brach, R. M., "Experiments on the Low-Velocity Impact of Microspheres with Planar Surfaces," *Aerosol Sci. Tech.* 23(1), 102-182 (1 Jul 1995).
- ¹⁸Oberle, L., and Landis, G., *Brief History of the NASA Lewis QCM Experiment*, JPL/NASA, available at <http://www.lerc.nasa.gov/WWW/OptInstr/QCMblurb.html> (accessed 1 Feb 1999).
- ¹⁹Oberle, L., and Landis, G., *Mars Pathfinder: Measurement of Dust Settling*, available at <http://mpfwww.jpl.nasa.gov/ops/mae.html> (accessed 1 Feb 1999).
- ²⁰Simon, C. G., Skrivaneck, R. A., Munzemer, R., Tuzolino, A. J., Tanner, W. G., et al., "The Orbital Debris Detector Consortium: Suppliers of Instruments for In-Situ Measurements of Small Particles in the Space Environment," in *Proc. LDEF-69 Months in Space: Third Post Retrieval Symp.*, Williamsburg, VA, pp. 1361-1377 (8-12 Nov 1993).
- ²¹Thompson, M. W., *Final Report for SM-3 Nose Cone Tests at JHU/APL*, AATDL-98-116, JHU/APL, Laurel, MD (Sep 1998).
- ²²Uy, O. M., and Cain, R. P., *Contamination Results Measured by Miniature Quartz Crystal Microbalances During SM-3 Nose Cone Wind Tunnel Test Series II*, TSM 98-106, JHU/APL, Laurel, MD (5 Oct 1998).
- ²³Mattix, M. P., and Linevsky, M. J., *SM-3 Nose Cone Contamination Analysis*, A1F (2) 98-U-084, JHU/APL, Laurel, MD (10 Aug 1998).
- ²⁴Nanonics, Corp. Home Page, available at <http://www.nanonics.com> (accessed 1 Feb 1999).

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